

Abstract

<i>Charles University in Prague</i> <i>Faculty of Pharmacy in Hradec Králové</i>	
Department of:	<i>Analytical chemistry</i>
Candidate:	<i>Bc. Lukáš Grebeníček</i>
Supervisor:	<i>PharmDr. Hana Sklenářová, Ph.D</i>
Title of Diploma Thesis:	<i>Verification of practical application of parallel SIA determination of inorganic anions in real samples of surface water</i>

This diploma thesis is divided into two parts. In the first one, the experimental determination deals with verification of practical application of chosen anions (SiO_3^{2-} , Cl^- , NO_2^- , NO_3^-) by sequential injection (SIA) assay followed by valid ISO norms for flow injection (FIA) assays. The second part compares determination of nitrites and nitrates by the SIA assay and comparative assay from MERCK company.

Determination of dissolved silicates was accomplished in accordance with the Czech technical norm ČSN EN ISO 16264. This technical standard for FIA assay is based on reaction of dissolved silicates with reagent solution. Reagent is composed of acidic solution of molybdenite, which reacts with silicates and phosphates generating molybdic-silicate acid and molybdic-phosphate acid. Molybdic-phosphate acid is decomposed by oxalic acid solution and molybdic-silicate acid is reduced by stannic chloride to molybdic blue – the final product of reaction – assayed spectrophotometrically at 710 nm.

Determination of chlorides by the valid Czech technical norm ČSN EN ISO 15682 is based on reaction of mercuric thiocyanate with iron nitrate. Thiocyanate ions (which were displaced by chlorides) reacts with Fe^{III} ions creating red ferriferous thiocyanate complex. Absorbance of this complex is measured at 465 nm.

For assessment of nitrites and nitrates cadmium reducing column was used which reduces nitrates to nitrites. Nitrates were determined together with nitrites as sum. Sample was mixed with chromogenic reagent containing sulphanilamide and naphthylethylene diamine. Final product generated by Grisse's reaction was measured at 545 nm.

In the first part of experiment mixed-solutions of listed anions were measured. Flow-cells with 10 mm, 20 mm optical length and capillary flow-cell (10 mm) were compared.

In the second part of experiment calibrations of nitrites, nitrates and repeatability of determination were measured. Calibration curves of NO_2^- concentration shown linearity in

the interval between 1-10 mg/l with correlation coefficient $R^2 = 0.992$. Calibration range in NO_3^- determination shown linear interval by $c = 5-50$ mg/l with correlation coefficient $R^2 = 0.9943$. In this part comparison of nitrite and nitrate assessment in samples obtained from surface water by the SIA assay and assay from MERCK company based on testing strips is also described. Results from both assays are comparable only with certain limitation given by assays sensitivities.

Determination of nitrites and nitrates confirms applicability of the SIA assay in routine analysis based on automatic system with possibility to analyse a large number of samples in short time.

